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Linseed oil as a potential resource for bio-diesel: A review

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ABSTRACT

The energy crisis contributed to the development of bio-diesel production. Petroleum, charcoal and natural gas sources are limited and will be exhausted by the next century. Thus, looking for alternative source of energy is of vital importance Vegetable oils are a renewable and potentially inexhaustible source of energy with an energetic content close to diesel fuel. In recent years, bio-diesel has become more attractive as an alternative fuels for diesel engine because of its environmental benefits and it is made from renewable resource. Since edible oil demand is higher than its domestic production; there is no possibility of diverting this oil for production of bio-diesel in India. Being a tropical country, India is rich in forest resources having a wide range of trees, which yield a significant quantity of oilseeds. India is importing crude petroleum & petroleum products from Gulf countries. Indian scientists searched for an alternate to diesel fuel to preserve global environment and to withstand economical crisis. This review paper describes the production of linseed oil, its properties, composition and future potential for bio-diesel. Linseed plant contains high amount of oil in its seeds which can be converted to bio-diesel. Fatty acid compositions of linseed reported in literature are provided in this review. In this study the properties of methyl ester of linseed oil are compared with the properties of fossil diesel. The objective of this review is to give an update on the linseed plant, the production of bio-diesel from the linseed oil and research attempts to improve the technology of converting linseed oil to bio-diesel and the fuel properties of linseed bio-diesel. The technological methods that can be used to produce bio-diesel are presented together with their advantage and disadvantages. Many other areas that need to be researched on linseed oil are pointed out in this review.

Contents

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1. Introduction

Fuel crisis and environmental concerns have renewed interest of scientific community to look for alternative fuels of bio-origin such as vegetable oil. Bio-diesel was found as the best alternate fuel, technically and environmentally acceptable and easily available. Bio-diesel consists of methyl/ethyl esters of fatty acids and is suitable for use in diesel engines [1]. It is an alternative diesel fuel, made from renewable biological sources such as vegetable oils and animal fats by transesterification with monohydric alcohols [2,3,4]. Bio-diesel is the name given to any diesel equivalent bio-fuel which can be used in any unmodified diesel engines vehicle [5]. The concept for producing fuel from vegetable oil for diesel engine is not radically new concept. The use of vegetable oils the inventor of the diesel engine Rudolph diesel first tested in 1900 that renewable resource material like peanut oil as a fuel in his compression ignition engine in place of petroleum diesel without any problem [2,6,7]. Mostly bio-diesel is prepared from many different feedstock oils like soybean, rapeseed, sunflower, safflower, etc., throughout the world these oils are essentially edible in nature [8–11]. Few attempts have been made for producing bio-diesel with non-edible oils like karanj, jatropha and linseed oil especially in India [8,9,12]. India and other developing countries can secure their energy dream with the production of renewable energy from non-edible vegetable oil such as linseed oil and jatropha [13]. The climatic and soil condition of our country is convenient for the production of linseed (*Linum usitatissimum*) crop [14]. The main production area for the linseed oil is in village [15]. Linseed plant contains high amount of oil in its seeds which can be converted to bio-diesel. Linseed is highly oleaginous (around 40%) [16]. This review highlights the efforts made to develop bio-diesel from linseed oil, which is available in India.

2. Methods of bio-diesel production

The problem of substituting vegetable oil for diesel engine as a fuel is mostly associated with high viscosity, low volatility and polyunsaturated characters. These can be changed in at least four different ways: Pyrolysis, Microemulsion, Dilution and Transesterification [17,18]. Bio-diesel can be prepared by different methods [19].

2.1. Transesterification

Transesterification can be catalyzed by acids, bases or enzymes. It is most common and popular method of bio-diesel production from vegetable oil [6,19]. It is refers to acid catalyst or base catalyst chemical reaction involving oil (triglyceride) and an alcohol (methyl/ethanol) to yield mono-alkyl esters of long chain fatty acid (bio-diesel) and glycerol [3,13]. The main factors affecting tranestericati on are the amount of alcohol, free fatty acid content, presence of water, and catalyst reaction, tem, pressure and time [20–22]. The performance of vegetable oil can be improved by modifying vegetable oil through transesterification process. The purpose of this process is to lower the viscosity of the vegetable oil [17]. Vegetable oil consists of three carbon backbones with long chain fatty acids and bio-diesel composed of fatty acid chains that are chemically bonded to one methanol molecule [23]. It is a chemical process; in this process triglyceride is reacted with an alcohol [24]. It is displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis except that an alcohol is used instead of water.the reaction can be represented as shown below: chemical reaction of synthesis of bio-diesel [10,24,25] Figs. 1–4.

2.1.1. Base catalyzed transesterification

Base catalysis method is the most popular way and most often used commercially for bio-diesel production. It is using for low fatty acid feed oil [23]. In this method, a triglyceride reacts with an alcohol in the presence of a catalyst such as strong base (KOH/NaOH), producing a mixture of fatty alkyl esters (known as bio-diesel) and glycerol [26]. Trancesterification of triglycerides by an alkaline catalyst also called alcoholysis. The base-catalyzed transesterification of vegetable oils much faster than the acid-catalyzed reaction [27]. Fangrui Maa and Hanna [28] studied that in the case of the alkali catalyzed reaction, the mechanism was formulated in three steps, in the first of them the anion of the alcohol will attack the carbonyl carbon atom of the triglyceride and a tetrahedral intermediate will be formed. The second step implies that intermediate reacting with an alcohol to regenerate the anion of the alcohol. Finally, in the third step, a rearrangement of the tetraheldral intermediate takes place, resulting in the formation of a fatty acid ester and a diglyceride [28,29].

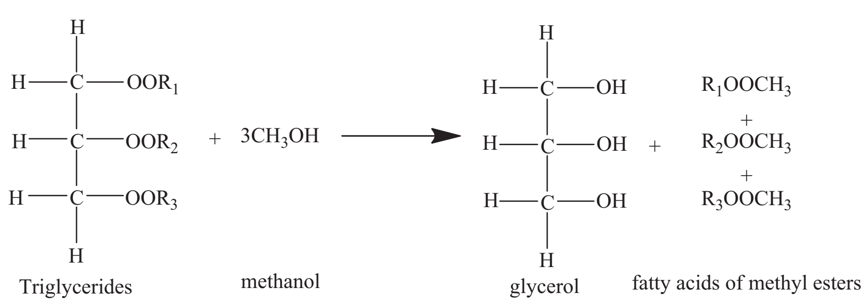

Fig. 1. Transesterification of vegetable oil [21,22,24,25].

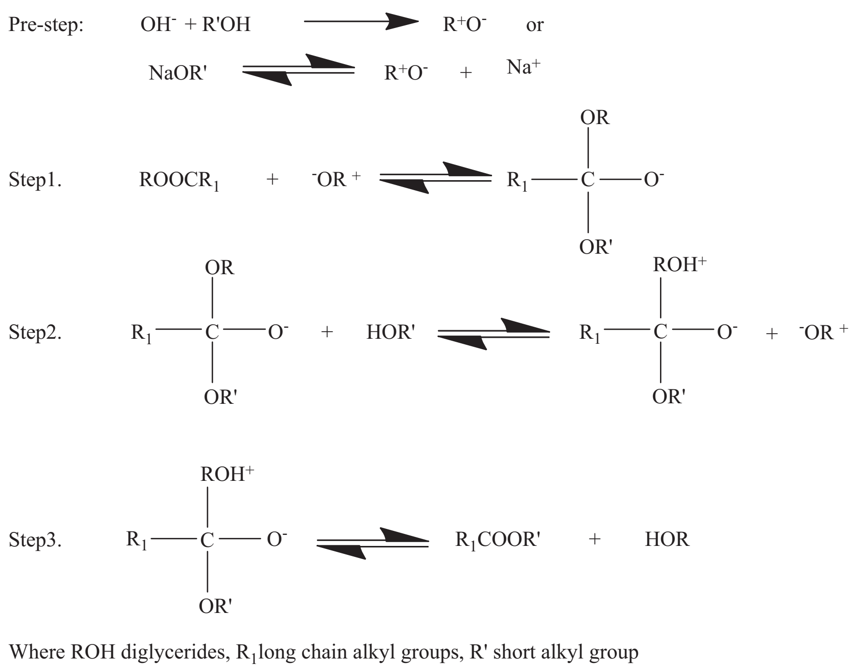

Fig. 2. The mechanism of the alkali catalyzed transesterification [28,29].

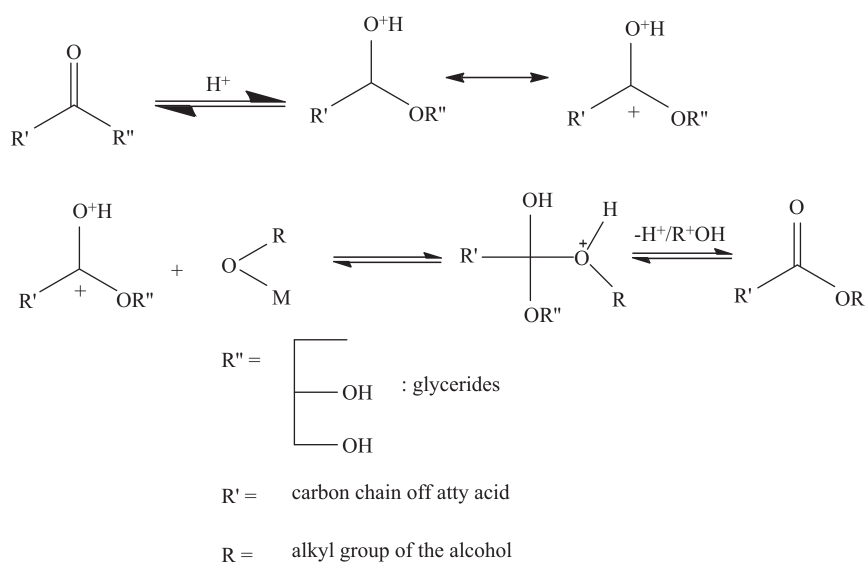

Fig. 3. Acid catalyzed transesterification [28,25,29].

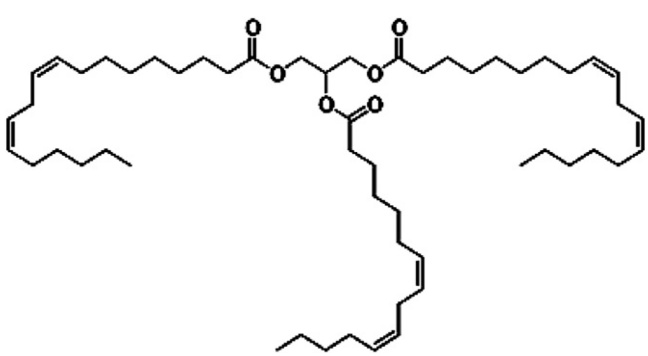

Fig. 4. Structure of linolein—a triglyceride[45].

2.1.2. *Acid catalyzed transesterification*

Acid catalyzed can be used for high free fatty acid containing bio-diesel feed oils like karanj and mahua oil or strong acid (H_2SO_4) [30,31]. The acid-catalyzed transesterification process reduces the high FFA content of the vegetable oil to about 2% FFA [30,32]. In this method free fatty acids are react with methanol in the presence of acid catalyst such as sulfuric acid [29,23,28,31].

2.1.3. *Enzymatic transesterification*

Haldar and Nag reported the production of bio-diesel through enzyme catalyzed transesterification by using Lipase enzyme and methanol [33]. Comparative to chemical approach enzymatic approach for bio-diesel production offers more advantages. For

Table 1
Chemical structure of common fatty acids [16,17,44].

Name of fatty acid	Chemical name of fatty acids	Structure	Formula
Palmitic	Hexadecanoic	C16:0	$C_{16}H_{32}O_2$
Stearic	Octadecanoic	C18:0	$C_{18}H_{36}O_2$
Oleic	Octadecenoic	C18:1	$C_{18}H_{34}O_2$
Linoleic cis-9, cis-12	Octadecadienoic	C18:2	$C_{18}H_{32}O_2$
Linolenic	Cis-9, cis-12, cis-15-octadecatrienoic	C18:3	$C_{18}H_{30}O_2$

reasons of easy product separation, easy glycerol recovery and the absence of side reactions [7,5,33].

$$R\text{-COOH} + \text{MeOH} \xrightarrow{\text{Lipase}} \text{R-COOMe} + H_2O$$

Resulted that bio-diesel was synthesized enzymatically with Novozym-435 lipase in presence of supercritical carbon dioxide. Rathore and Madras [18]. Enzyme that was found to be capable of catalyzing transesterification is lipase. Lipase can be obtained from microorganisms [6]. The main disadvantage of enzyme catalyst process stems from the high cost of the lipases as catalyst [34].

2.1.4. Supercritical and subcritical alcohol transesterification

An alternative, catalyst-free process for transesterification uses supercritical methanol. The purification becomes much simpler, and it produces higher yield [35,36]. In this process in the feedstock; free fatty acids are converted to methyl esters instead of soap, so a wide variety of feedstock's can be used. Also the catalyst removal step is eliminated [37]. Energy costs of production are similar or less than catalytic production routes [36].

3. Current status—Linseed oil

Linseed oil, its scientific name is *Linum usitatissimum*, belongs to the family Linaceae and the genus *Linum* which has 100 species. It originated from Mediterranean coastal countries and is cultivated in Canada, Argentina, India and USA [38,39]. It is an annual species of the *Linaceae* family, growing to a height of 0.3–1 m, which is cultivated for the production of textile fiber, seed and linseed oil. Linseed is cultivated in Araucanía Region for oil extraction. Studies have shown that crop yield is higher in this Region, because of its soil and climate characteristics. Linseed is best suited for fertile, fine textured and loamy soils: an important factor is the amount of rainfall during the growing period. Adequate moisture and relatively cool temperatures, particularly during the period from flowering to maturity, seem to favor both oil content and oil quality. The seed is located in the extremities of the branches in round capsules, each of which contains from one to ten seeds [38,40,41]. In India, it is grown mainly for seeds, used for extracting oil. Under optimum conditions the oil content of linseed seeds varies from 33 to 47%. In India various states have sufficient forest area for the plantation of linseed. Madhya Pradesh leads in yield and acreage, followed by Uttar Pradesh and Maharashtra, Bihar, Rajasthan, Karnataka and West Bengal also grow linseed in large areas. Madhya Pradesh and Uttar Pradesh together contribute to the national linseed production to the extent of about 70%. India accounts for about 1.9 million hectares, with a seed production of 4.98 lakhs of tonnes, the crop in northern India generally gives higher yield than in central and peninsular India. The irrigated crop may yield 1200 to 1500 kg per ha. and occupies the third rank among the linseed-producing countries Australia and Canada [39]. Linseed oil is the most commonly used carrier in oil paint. It is available in Asian countries. It is an important oilseed in the world [6]. Linseeds are a source of high quality proteins, soluble fiber and a high content of polyunsaturated fatty acids.They present values of 30–40% lipids, 20–25% proteins, 4–8% moisture, 3–4% ash and 20–25% dietary fiber [8].

3.1. Chemical composition of linseed oil

The chemical composition of linseed oil varies with geographical location and variety. Oils from different sources have different fatty acid compositions [17]. Linseed oil consists chiefly of three glycosides, linolein, linolenin, and olein. A small amount of free fatty acids, such as palmitic and arachidic, is also present [42]. The triglycerides of linseed oil contain five major fatty acids [43]. The main fatty acids, which are commonly found in the linseed oil, were palmitic, stearic, oleic, linoleic and α-linolenic Table 1 summarized the fatty acid composition of linseed oil [16,17,44]. It was observed that percent of FFAs in linseed is 1.94% Tiwari and Kumar [13].

Linolein, which is present in linseed oil to the extent of about 20%, is the glyceride of linoleic acid, and has the formula $C_{57}H_{98}O_6$ [42,45].

3.2. Cake/meal utilization

After extraction of the linseed oil a large quantity of cake remains. The linseed cake is mainly used as a cattle feed, it is a very good manure and animal feed [39]. Linseed cake/meal is used as an additive in baking products [46]. Linseed meal is used normally for ground un-extractracted seed (35%), ground linseed cake (10% oil) and linseed meal (3%) from a solvent plant. Rich oil content can affect texture and flavor of meat and butter obtained [40].

3.3. Protein, gluten and carbohydrates

Amino acids are the building blocks of protein (20–25%). Pattern of linseed protein is similar to that of soybean protein. Linseed is gluten-free and it is low in carbohydrates [47].

3.4. Fibers

Linseed straw produces fiber of good quality and fiber accounts for about 28% of the weight of linseeds fat [8,47]. It was most popular plant used to produce fibers for linen in Egypt [38] reported that two main varieties of linseed, one gives a high yield of seed and other fiber [40,41]. Fiber occurs as structural material in the cell walls of plants and has important health benefits for humans and it is also used in paper making [47].

3.5. Phenolics, phenolic acids, flavonoids and lignans

Phenolics are plant compounds that have many different functions, including adding color to the plant and attracting bees and other insects for pollination. Linseed contains at least three types of phenolics: phenolic acids, flavonoids, and lignans. Linseed contains about 8 to 10 g of total phenolic acids per kilogram of linseed and about 35–70 mg of flavonoids/100 g. It is a very rich source of a lignan [47].

4. Extraction of crude linseed oil

Linseed oil is obtained from linseed. It is obtained by various methods including pre-expelling, and hexane extraction of the press cake [48]. The yellowish drying oil is derived from dried ripe seeds of linseed plant through pressing and extraction. It is available in varieties such as cold pressed, alkali refined, sun bleached, sun thickened, and polymerized (stand oil) marketed as linseed oil [6]. The seeds are pro-pressed and solvent extracted to obtain oil. As the seed itself is very hard, it must be crushed or

softened by boiling before being fed. Both gums and waxes occur in the crude oil, settling in tanks for upto three weeks allows 1% of the oil to separate. Subsequently if the decanted oil is warmed rapidly to 110 °C a further deposit of 0.5% takes place. Such treatment is long established and described as a break of the oil. Further degumming, neutralisation and washing may be based on the refining of oil where the difficulties are comparable [47]. Supercritical carbon dioxide (SC–CO_2) and organic solvent extraction were used to extract linseed oil. Galvao et al. was performed a comparative analysis between supercritical CO_2 extraction (SC–CO_2) and organic solvent extraction (OS). Extraction yields using OS were higher than those obtained with the SC–CO_2 technique [16].

5. Linseed oil for bio-diesel production

Linseed has excellent potential for byproduct development, and it has a high level of oil. Under optimum conditions linseed seeds can yield up from 33–47% oil content. Modification to the fatty acid profile and increased yield would make it an ideal candidate for use within the bio-diesel industry. And the climatic and soil condition of our country is convenient for the production of linseed (Linum usitatissimum) crop [14]. Major problems encountered with linseed oil as bio diesel used in CI engine are its low volatility and high viscosity due to long chain structure. The common problems faced are excessive pumping power, improper combustion and poor atomization of fuel particles. The conversion of the vegetable oil as a CI engine fuel can be done any of the four methods; pyrolysis, micro emulsification, dilution/blending and transesterification [42]. Methyl ester of linseed oil produced from transeterification process by using methanol and potassium hydroxide (KOH) as a alkali-catalyst [14,15,45,49,]. Methyl, ethyl, 2-propyl and butyl esters were made from linseed oil through basic catalyst (KOH/NaOH) transesterification. The viscosity of linseed oil bio-diesel highly decreases after transesterification process [50,51]. Linseed bio-diesel produces by transesterification process and the properties of linseed oil, bio-diesel are comparable with conventional diesel fuels. Higher yield 88% bio-diesel was found at 20% methanol, 0.5% NaOH and 55 °C reaction temperature. The maximum bio-diesel production measured after 15 h reaction time [14]. Investigated that combustion and emission performance of linseed oil and linseed oil methyl ester in a stationary single cylinder, four stroke diesel engine and compared it with baseline data of diesel fuel. Agarwal and Agarwal [52] reported that enzymatic synthesis of linseed oil methyl ester from linseed oil supercritical alcohol and supercritical carbon dioxide and it is a most promising method to replace the catalyst transesterification process. High yield was obtained at a short time at supercritical conditions and the conversion increases with molar ratio of alcohol to oil up to 40:1 Mahesh and Giridhar [49,53]. Demirbas was also reported methyl & ethyl ester as bio-diesel fuels were made from linseed oil transesterification in non-catalytic super-critical fluids condition. The transesterification of linseed oil in supercritical fluids such as methanol and ethanol has proved to be the most promising process [50] reported methyl ester produced from linseed oil by using transesterification double step process (TDSP) and eliminats soap or emulsion formation problems. TDSP involves two step transesterification process with a basic catalyst included a reduction in the catalysts concentration and reaction time of the first step and modified to bio-diesel yield 98% for linseed oil Guzatto et al. [54]. The fatty acid compositions (wt%) of linseed bio-diesel are shown in Table 2 [51].

Table 2
Fatty acid compositions (wt%) of linseed biodiesel [51].

Fatty acid	Linseed methyl ester	Linseed ethyl ester	Linseed 2-propyl ester	Linseed Butyl ester
Palmitic (16:0)	5.2	5.1	5.1	5.3
Stearic (18:0)	3.2	3.1	3.1	3.2
Oleic (18:1)	14.5	13.7	14.2	13.8
Linoleic (18:2)	15.3	15.2	15.1	15.2
Linolenic (18:3)	61.9	62.9	62.5	62.6

6. Fuel properties of linseed bio-diesel

Properties of linseed oil methyl ester are comparable with diesel fuel [14,52]. Fuel properties of methyl, ethyl, and 2-propyl and butyl esters of linseed oil shown in Table 3 [50,51].

The viscosity and flash point values of linseed oil methyl and ethyl esters highly decrease after transesterification process [17]. Evaluated that chemical properties of linseed ester fuel, 0.03% free fatty acids, peroxide value 27.8 meq/kg and 0% free glycerol [55]. Agrawal and Kumar (2008) reported that, after transesterification process the density of linseed oil and linseed oil methyl ester has become almost similar density as that of conventional diesel fuel and heating value of linseed oil is about 10% lower than diesel due to higher oxygen content in it [56].

7. Engine performance and emission parameters of linseed oil and its blends

Agrawal investigated the use of linseed bio-diesel in compression ignition engines found to develop a very compatible engine fuel system with lower emission characteristics [15].

Table 3
Fuel properties of linseed oil alkyl esters [50,51].

Properties fuel	Linseed methyl ester	Linseed ethyl ester	Linseed 2-propyl ester	Linseed butyl ester
Density at 25 °C (g/ml)	0.887	0.884	0.888	0.877
Dynamic viscosity at 400 °C (10^{-4} Pa s)[a]	3.32	3.64	4.88	4.06
Acid value (mg KOH/g)	0.335	0.324	0.586	0.254
Cloud point (°C)	0	−2	3	−10
Pour point (°C)	−9	−6	−12	−13
Gross heat of combustion Hg (MJ/kg)	40	39.65	39.56	40.38
Volatilization (°C)	176.1	178.4	178.7	173.2
Higher heating value (MJ/kg)	40	39.65	39.56	40.38
Distillation temperature (°C)	188.8	191.9	192.6	197.1

Table 4
Fatty acid composition and natural antioxidants in linseed methyl ester[45,61].

Linseed methyl ester	Fatty acids			Content of natural antioxidants
	Saturated	Unsaturated	Polyunsaturated	Tocopherols and tocotrianols, (mg/kg)
	4.4	25.1	70.1	694

7.1. CO and NO_x emission characteristics for linseed oil bio-diesel

Most researchers found a decrease in CO emissions when substituting diesel fuel with bio-diesel [44]. Carbon emission depends upon combustion efficiency and carbon content of the fuel. Methyl ester of linseed oil has lower carbon-monoxide emission compared to diesel and 100% linseed oil [57]. Evaluated that impact of oxidized linseed bio-diesel–diesel blends on engine performance and emission due to presence of extra oxygen in linseed bio-diesel additional oxidation reaction take place between oxygen and carbon-monoxide emission [14]. Carbon monoxide emission of blends reduced due to increasing in oxygen content and the extra oxygen content of bio-diesel promotes complete combustion of fuel and lower particulate emission [52,58]. Studies the operation of test engine with linseed oil-diesel fuel blends fuel a wide range of engine load condition was shown that NO*x* level is higher for diesel–bio-diesel blends than conventional diesel fuel [14,57]. Also investigated brake specific fuel consumption (BSFC) for 10%, 20%, bio-diesel–diesel blends is similar to conventional diesel fuels Nurun and Najmul [14]. Properties of bio-diesel, lower heating value, higher density and higher viscosity play primary role in engine fuel consumption for bio-diesel [58]. Investigated that methyl ester of linseed oil has higher exhaust gas temperature compared to diesel and lower compared to pure linseed oil [57].

7.2. Oxidation stability for linseed oil

Oxidation stability [59] is an important fuel quality parameter of bio-diesel [60]. The auto-oxidation process takes place during the storage of fatty acid methyl esters. In the primary stage of oxidation process peroxides and hydro-peroxides are formed. Later, aldehydes and ketones are formed and finally during the polymerization process resins are produced [61,48]. Fatty acid methyl esters are more sensitive to oxidative degradation than conventional diesel fuel, due to their chemical composition, esters of vegetable oil content high concentration of unsaturated fatty acid [62]. The oxidation rate of fatty acid methyl ester depends on the nature of fatty acids of feed oil, temperature reaction catalyst and inhibitors contained in fats [60]. Linseed oil is also rich in unsaturated fatty acid content. The results of high degree un-saturation in linseed oil are elevated reactivity and lower oxidative stability [54]. Reported that linseed oil esters have better stability compared to the linseed oil as far FFA increasing is concerned [17]. Antioxidants are chemicals that inhibit the oxidation process and it is contributed to the stabilization of the lipid sample [45,63]. In vegetable oils and their esters, antioxidants can come from two sources: natural antioxidants and added synthetic antioxidants [63]. Oxidation stability of fatty acid methyl esters of linseed oil can be improved by using synthetic antioxidants [61]. Tocopherols are optimized with respect to antioxidant capability of vegetable oil. It is a phenolic compound that occur naturally in vegetable oils [63,64]. Fatty acid composition and content of natural antioxidants present in fatty acid methyl ester of linseed oil shown in Table 4 [45,61].

7.3. Thermal stability for linseed bio-diesel

Thermal efficiency is the true indication of the efficiency with which the chemical energy input in the form of fuel is converted into useful work [65]. The thermal efficiency of the engine was found to improve by increasing concentration in the blend. It was observed that all bio-diesel blends have thermal efficiency higher than conventional diesel [52]. Brake thermal efficiency for 25% blend of linseed oil with diesel is very close to that of diesel fuel but Avinash Kumar was reported 50% linseed oil blend found more thermal efficient than other blends [52,56,57]. Linseed oil are also rich in unsaturated fatty acid content, but especially in lineleic acid and linolenic acid most of the vegetable oils contain higher amount of poly-unsaturated fatty acids it, tends to be vulnerable to thermal degradation. Linseed bio-diesel was produced by supercritical methanol process and its degradation behaviors was studied in supercritical methanol by exposing fatty acid methyl ester found that polyunsaturated methyl esters such as methyl linoleate and methyl linolenate were partly degraded and isomerized into trans-type with a rise in temperature above 300 °C these changes were likely to occur for poly unsaturated fatty acids with higher degree of un-saturation [45,48,49]. Investigated that thermal efficiency of the engine at different bio-diesel blends is almost similar to diesel [8]. It was studied that possible reason for improved thermal efficiency may be more complete combustion of bio-diesel and its lubricity due to higher oxygen content in it [52,56]. Jaichandar and Annamalai studied high sulfur diesel blended linseed oil bio-diesel was tested in a single cylinder 4 kw portable engine and showed increase in thermal efficiency [65].

8. Conclusion

Very few researchers concentrated on non-edible oil for the bio-diesel production. Non-edible oil can also be utilized for making bio-diesel fuel as edible oil is heavy demand for cooking purpose. The main advantage for fuel production from non-edible oils reduces the use of petroleum diesel. Linseed oil is non-edible oil. In India, it is grown mainly for seeds, used for extracting oil. Under optimum conditions the oil content of linseed seeds varies from 33 to 47%. Linseed plant is traditionally cultivated for the production of textile fiber. It is also a high source of oil which can be converted into bio-diesel and provide a major source of renewable energy both locally and inter-nationally. There is need to investigate an appropriate method for the transesterification of *Linum usitatissimum* oil based on its properties. The fuel properties of linseed bio-diesel are comparable to those of fossil diesel. Linseed oil has lower oxidative stability and linseed oil esters have better stability compared to the linseed oil. There is need to research on oxidative stability of linseed oil. It is a promising source of bio-diesel.

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